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METHODS OF CHEMICAL ANALYSIS OF COPPER-PHOSPHORUS BRAZING ALLOYS

PART II DETERMINATION OF PHOSPHORUS

UDC 669·35' 779 : 543·847 : 621·791·36

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Rs 5·00

Gr 12

August 1976

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-PHOSPHORUS BRAZING ALLOYS

PART II DETERMINATION OF PHOSPHORUS

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-PHOSPHORUS BRAZING ALLOYS

PART II DETERMINATION OF PHOSPHORUS

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 23 June 1976, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Chemical analysis of copper-phosphorus brazing alloys, the chemical composition of which is specified in IS:2927-1975*, is covered in two parts of this standard. In this part, method for determination of phosphorus in copper-phosphorus is covered. The methods for determination of silver and copper are given in Part I of this standard.

0.3 In the formulation of this standard due weightage has been given to the need for international co-ordination among the standards and practices prevalent in different countries in addition to relating it to the practices being followed in the field in this country. This has been achieved by deriving assistance from '1972 Book of ASTM Methods for chemical analysis of metals, Part 32', issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard (Part II) prescribes method for chemical analysis of phosphorus in various grades of copper-phosphorus brazing alloys as specified in IS:2927-1975*.

2. SAMPLING

2.1 Laboratory Sample — It shall be drawn and prepared in accordance with IS: 1817-1961‡.

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*Specification for brazing alloys (*first revision*).

†Rules for rounding off numerical values (*revised*).

‡Methods of sampling non-ferrous metals for chemical analysis.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis

4. DETERMINATION OF PHOSPHORUS

4.1 Outline of the Method — The sample is dissolved in concentrated nitric acid and phosphorus is precipitated with ammonium molybdate. The precipitate is dissolved in ammonia and re-precipitated, dried and ignited to constant mass.

4.2 Reagents

4.2.1 *Concentrated Nitric Acid* — rd = 1·42 (conforming to IS : 264-1968†).

4.2.2 *Potassium Permanganate Solution* — 6 percent $\left(\frac{m}{v}\right)$.

4.2.3 *Potassium Nitrite Solution* — 2 percent $\left(\frac{m}{v}\right)$.

4.2.4 *Ammonium Nitrate* — solid.

4.2.5 *Ammonium Molybdate Solution* — Add solution (A) (*see* **4.2.5.1**) slowly and with constant stirring to solution (B) (*see* **4.2.5.2**) kept cool in a cold water-bath. Add 10 ml of ammonium phosphate (one gram per litre) and keep the solution at least for 24 hours. Filter the solution through Whatman filter paper No. 1 before use.

4.2.5.1 *Solution (A)* — Dissolve 100 g of molybdic acid (MoO_3, 85 percent) or 118 g of ammonium molybdate in a mixture of 145 ml of concentrated ammonium hydroxide and 270 ml of water. Cool the solution.

4.2.5.2 *Solution (B)* — Add 490 ml of concentrated nitric acid to 1 150 ml of water and cool.

4.2.6 *Dilute Nitric Acid* — 2:98 (*v/v*) and 1 percent (*v/v*).

4.2.7 *Dilute Ammonium Hydroxide Solution* — 1 : 1 (*v/v*) and 5 percent (*v/v*).

4.2.8 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

4.2.9 *Magnesia Mixture* — Dissolve 55 g of magnesium chloride ($MgCl_2.6H_2O$) and 70 g of ammonium chloride in 650 ml of water. Dilute to one litre with 50 ml of dilute ammonium hydroxide [25 percent (*v/v*)].

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*Specification for water, distilled quality (*revised*).

†Specification for nitric acid (*first revision*).

4.2.10 *Concentrated Ammonium Hydroxide* — rd = 0·90.

4.2.11 *Dilute Ammoniacal Ammonium Nitrate Solution* — 10 percent $\left(\frac{m}{v}\right)$.

4.3 Procedure

4.3.1 Transfer 0·5 g of an accurately weighed sample into a 400-ml conical flask. Dissolve in 25 ml of concentrated nitric acid, heat gently to expel brown fumes, and add 100 ml of water. Heat the solution to boiling and add a saturated solution of potassium permanganate drop by drop until a deep purple colour persists. Boil for 5 minutes, then destroy the excess potassium permanganate and precipitated manganese dioxide by adding potassium nitrite solution drop by drop until the solution is of clear blue colour. Boil for 5 minutes to expel nitrous fumes.

4.3.2 Cool the solution to 60°C. Neutralize with dilute ammonium hydroxide solution (1 : 1), add 5 ml of concentrated nitric acid, and add 5 g of ammonium nitrate. Add 60 ml of the ammonium molybdate solution in small portions, while swirling the flask. Close the flask with a rubber stopper, shake vigorously for 5 minutes, and allow to stand at room temperature for one hour. Filter through a Whatman filter paper No. 40 with pulp, wash the flask and precipitate 3 times with dilute nitric acid (2 : 98) and then wash 5 times with dilute nitric acid (1 percent).

4.3.3 Dissolve the precipitate in 20 ml of ammonium hydroxide solution (1 : 1). Wash filter paper with dilute ammonium hydroxide solution (5 percent). Collect the solution in 250-ml beaker. Acidify with dilute hydrochloric acid and cool to 15°C. Make just ammoniacal and add 10 ml of magnesia mixture with constant stirring, add ammonium hydroxide solution (5 percent) drop by drop till precipitation starts. Stir vigorously for 3 minutes and add 20 ml of concentrated ammonium hydroxide (total volume of the solution at this stage should not exceed 150 ml). Allow to stand for 4 hours.

4.3.4 Filter the precipitate through Whatman filter paper No. 42. Wash with dilute ammoniacal ammonium nitrate solution. Dry the precipitate alongwith the paper and transfer the precipitate to a previously weighed silica crucible. Incinerate the paper separately and transfer to the same crucible. Ignite slowly at 1 050°C to constant mass.

4.4 Calculation

$$\text{Phosphorus, percent} = \frac{A \times 0{\cdot}2787}{B} \times 100$$

where

A = mass in g of magnesium pyro-phosphate ($Mg_2P_2O_7$), and

B = mass in g of the sample taken.

INDIAN STANDARDS
ON
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IS:

964-1956 Silver solder
998-1959 Soft solder
999-1959 Brazing solder
4354 (Part I)-1967 Magnesium-aluminium brazing alloys: Part I Aluminium, manganese, zinc and silicon
4548 (Part I)-1967 Copper-gold brazing alloys: Part I Gold and copper
4646 (Part I)-1968 Chemical analysis of copper-phosphorus brazing alloys: Part I Silver and copper
4667 (Part I)-1968 Silver-copper brazing alloys: Part I Silver and copper
4667 (Part II)-1969 Silver-copper brazing alloys: Part II Silver, copper and tin
4703-1968 Silver-manganese brazing alloys

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Printed at Printograph, New Delhi, India